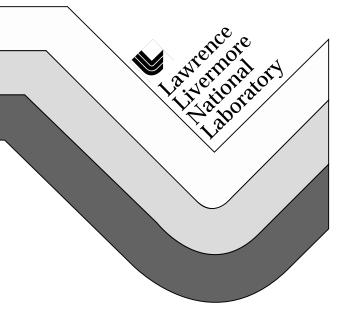
ARTI/MCRL Project Report Products of Motor Burnout (Second Quarter Report)

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January 15, 1995

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Work performed under the auspices of the U.S. Department of Energy by the Lawrence Livermore National Laboratory under Contract W-7405-Eng-48.



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DOE/CE/23810-48E

Summary:

The OSP (Operating Safety Procedure) required for performance of electrical arc testing of CFC replacement fluids was renewed. Electrical breakdown tests at one (1) atmosphere pressure have been performed for R-22, R-134a, and R-125/R-143a (50:50 blend; AZ-50), and breakdown products identified. No differences in HCFC breakdown products are seen in the presence or absence of lubricant oils. The design of the high pressure-high temperature test stand has been finalized, and construction initiated during this quarter. Three motor stators and rotors were received from Tecumseh Products Company for use in motor burnout tests. A test plan for the motor breakdown tests is in preparation.

Task Areas:

Task 1 - Literature Search

We have continued to monitor the literature for references related to CFC degradation/breakdown by thermal and/or electrical mechanisms. As breakdown products are identified from electrical discharge tests, we are monitoring the literature for confirming experimental evidence of their formation during CFC breakdown, as well as possible mechanistic explanations for the reactions leading to their formation.

Task 2 - Identification of Motor Burnout Products Atmospheric Pressure/Ambient Temperature Tests

R-22, R-134a and AZ-50 (R-125/R-143a blend) have all been subject to electrical breakdown testing at atmospheric pressure at ambient temperature, and the resulting breakdown products analyzed by GC (gas chromatography) with flame ionization detection (FID) and gas chromatography/mass spectrometry (GC/MS). Initial efforts at quantitation of the breakdown products by FID have proven to be difficult due to substantial variations in FID response for halogenated compounds. Although quantitation by GC/MS is more difficult, the GC/MS has proven to give much more stable response for the compounds identified. Quantitation is performed through the use of standards containing decane, hexafluorobenzene, 1,1,2-trichloro-1,2,2-trifluoroethane difluorochloromethane at varying concentrations. Identification of breakdown products is performed by comparison of mass spectra obtained with mass spectra contained in the following mass spectral libraries:

a. The Wiley/NBS Registry of Mass Spectral Data, F. W. McLafferty and D. B. Stauffer, John Wiley & Sons, New York, 1989.---112,300 compounds

- b. Mass Spectral Library: DuPont Fluorochemicals, personal communication from Pat Kaczarel, February 11, 1991.---126 compounds
- c. The Eight Peak Index of Mass Spectra, 4th Ed., The Mass Spectrometry Data Centre, The Royal Society of Chemistry, Thomas Graham House, Science Park, Cambridge, UK, 1991.---81,123 compounds
- d. NIST/EPA/NIH Mass Spectral Database, PC Version 4.0, US Department of Commerce, NIST, Gaithersburg, MD, May 1992. >100,000 compounds.

Initial arc testing was performed using 20kV, pulsed breakdown at 100 Hz pulse repetition frequency (prf), with an electrode gap spacing of 0.025" (25 mils) and a pulse width of 30 μ sec. For all fluids, electrical breakdown occurred at approximately 7 kV with an energy deposition of approximately 1.4 milli-joules/pulse. Fluids were subjected to electrical breakdown tests in the presence and absence of lubricant (Calgon C4 mineral oil for R-22; EM Karate RL32S polyolester oil for R-134a and AZ-50). No difference in volatile compounds formed was seen with or without lubricant present.

Table 1 lists the compounds tentatively identified to date by GC/MS in the 100,000 arc samples of R-22, R-134a and AZ-50. Compounds are listed in approximate elution order. The relative amounts formed (low, med., high) are also given in Table 1, where low signifies detection at concentrations less than 0.1% (w:w), med. means detection at concentrations between 0.1% and 1% (w:w), and high are those compounds formed at levels greater than 1% (w:w). Because exact isomer identification is not always possible in GC/MS analysis, isomer identification should be considered as tentative. Where specific isomers are listed, these were the primary mass spectral match as identified by the library search algorithm used. We are currently gathering boiling point data to assist in confirmation of isomer identification, where possible.

Elevated Pressure/Temperature Tests

The design of the elevated temperature/pressure test cell has been finalized, and a test stand is being constructed and is nearing completion. A sketch of the final elevated pressure test stand design is attached as Figure 1. The data acquisition system is being modified to allow continuous monitoring of pressure and temperature during the elevated pressure/temperature electrical breakdown tests.

Motor Burnout Tests

Three sets of motor components (stators and rotors) were received from Tecumseh Products Company for use in motor burnout tests. The components are for 4 hp motors of the type typically used as hermetically sealed motors on 2-3 ton commercial refrigeration units. A motor test bed is being prepared in Building 141. Three motors will be installed and charged with the fluids under testing (one per motor). We will be able to initiate and monitor breakdown at any point in the AC cycle for a controlled duration. Samples of fluid will be taken at regular intervals for analysis by GC/MS. A test plan is currently being prepared which will address the experimental details.

Work for Third Quarter (January-March, 1995)

We will complete replicate electrical breakdown tests at ambient pressure/temperature, and evaluate method reproducibility. Elevated pressure and temperature tests are expected to begin late January to early February. Motor burnout tests will be initiated as soon as the motor test bed is constructed and the test plan is finalized; motor burn-out tests may take place in parallel with elevated pressure/temperature tests. As the identification of compounds formed during electrical breakdown is finalized, we will monitor the literature to identify compatibility issues.

We will continue to monitor the literature for publications related to CFC replacements and specific issues of breakdown processes and products.

Table 1 Compounds Identified in Ambient Pressure/Temperature Electrical Breakdown Tests

	Approx. Concentration		
	- -		R143a/R-125
Compound ID	R-22	R-134a	(AZ50)
hexafluoroethane		high	high
tetrafluoroethene		high	high
1,1-difluoroethene		high	high
pentafluoroethane (R-125)			n/a
1,1,2,3,3,3-hexafluoro-1-propene		high	
trifluoroethene		high	
1,1,1-trifluoroethane (R-143a)			n/a
octafluoro-2-pentyne		low	
1,1,1,2-tetrafluoroethane (R-134a)		n/a	high
1,1,3,3,3-pentafluoro-1-propene			high
3,3,3-trifluoro-1-propyne			med
hexafluorocyclobutene			med
chlorodifluoromethane (R-22)	n/a		
1-chloro-1,1,2,2-tetrafluoroethane	med		
2-chloro-1,1,3,3,3-pentafluoro-1-propene	med		
3-chloro-1,1,2,3,3-pentafluoro-1-propene	med		
hexafluorocyclopentadiene isomer #1		low	med
hexafluorocyclopentadiene isomer #2		med	med
1,2-dichloro-1,1,2,2-tetrafluoroethane	high		
dichlorotetrafluoroethane	high		
1-chloro-3,3,3-trifluoropropyne	med		
hexafluorohexa-2,4-diyne		med	med
unidentified		low	med
4-(difluoromethylene)-2,3,3-			med
trifluorocyclobutene		_	
octafluoro-1,3,5-hexatriene		low	
chlorohexafluoropropane	med		
1,3-dichloro-hexafluoropropane	med		
1,2-dichloro-hexafluoropropane	med		
C5F5H		low	med
1,1-dichloro-2,2-difluoroethene	med		
1,2-dichloro-1,2-difluoroethene isomer #1	med		
1,2-dichloro-1,2-difluoroethene isomer #2	med		
trichlorofluoromethane	med		

Table 1 (con't) Compounds Identified in Ambient Pressure/Temperature Electrical Breakdown Tests

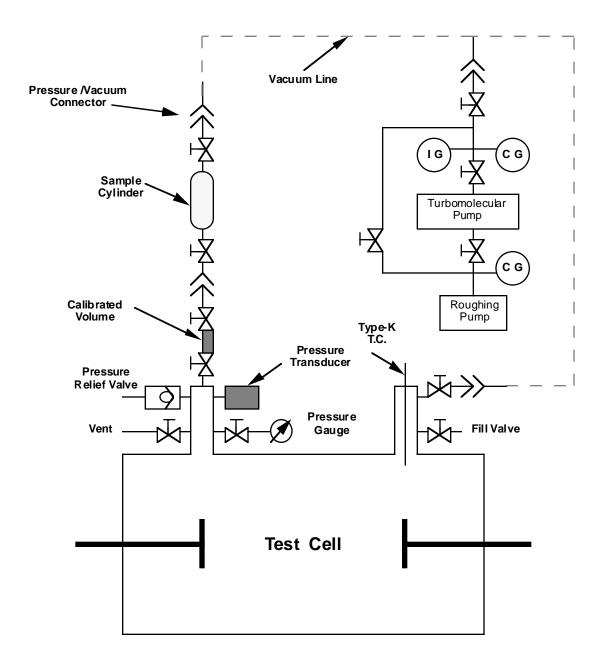
Approx. Concentration

	rippiox. Concentration		
			R143a/R-125
Compound ID	R-22	R-134a	(AZ50)
chloropentafluoroethane	med		
1,2-dichloro-1,3,3,3-tetrafluoro-1-propene isomer #1	low		
1,2-dichloro-1,3,3,3-tetrafluoro-1-propene isomer #2	low		
dichloroethyne	med		
C4F3Cl	low		
trans-1,3,3-trichloroprop-1-ene	med		
hexafluorochlorobutene	med		
chloropentafluoroethane	med		
trichloropropene	low		
1,2-dichlorotetrafluorocyclobutene isomer #1	med		
1,2-dichlorotetrafluorocyclobutene isomer #2	low		
1,2-dichlorotetrafluorocyclobutene isomer #3	low		
1,2,2-trichloro-1,1,3,3,3,-pentafluoropropane	low		
chloropentafluoroethane	med		
trichlorofluoroethylene	med		
chloropentafluorobenzene	low		
tetrachloroethane	low		
tetrachlorodifluoropropene	low		
dichlorotetrafluorobenzene	low		

Compounds listed in retention time order Approximate concentration ranges:

 $low - < 0.1\% \ by \ weight$ $med - between \ 0.1\% - 1\% \ by \ weight$ $high - > 1\% \ by \ weight$

Figure 1. Schematic of High Pressure/High Temperature Test Stand



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